(12) UK Patent Application (19) GB (11) 2 037 200 A

(21) Application No 7942220
(22) Date of filing 6 Dec 1979
(30) Priority data
(31) 969130
(32) 13 Dec 1978
(33) United States of America (US)
(43) Application published 9 Jul 1980
(51) INT CL[3] B22D 25/06
(52) Domestic classification B3F 11B 11C 11Y6 13A6 13A6C3 13A6D1C 13A6D3B 16A166 16B1 16B4
(56) Documents cited
GB 1426805
GB 1369270
GB 1285819
GB 1275030
GB 1158721
GB 1154844
GB 893528
(58) Field of search B3F

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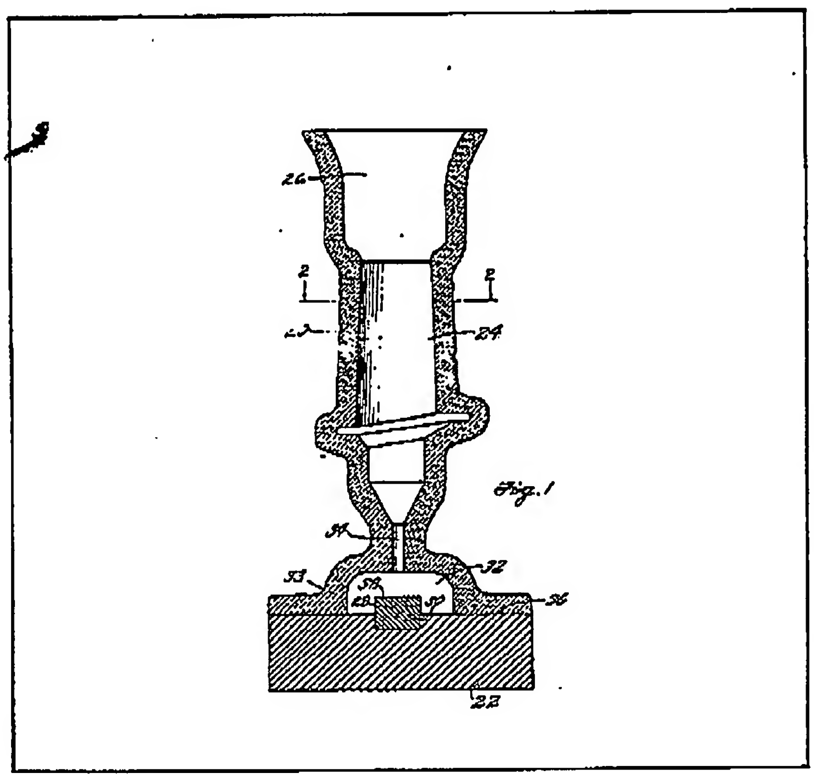
(54) Epitaxial solidification

(57) The starter section 32 of a directional solidification mold 20 contains a seed 28. Molten metal is flowed over and about the seed 28 to heat and partially melt it. A selector section 34 of the mold has reduced cross section compared to the starter section so that only epitaxially solidified metal will be formed in the article section.

A barrier layer 40, preferably of ceramic material, resistive to molton metal is preferably applied to portions of the seed to facilitate its removal and reuse.



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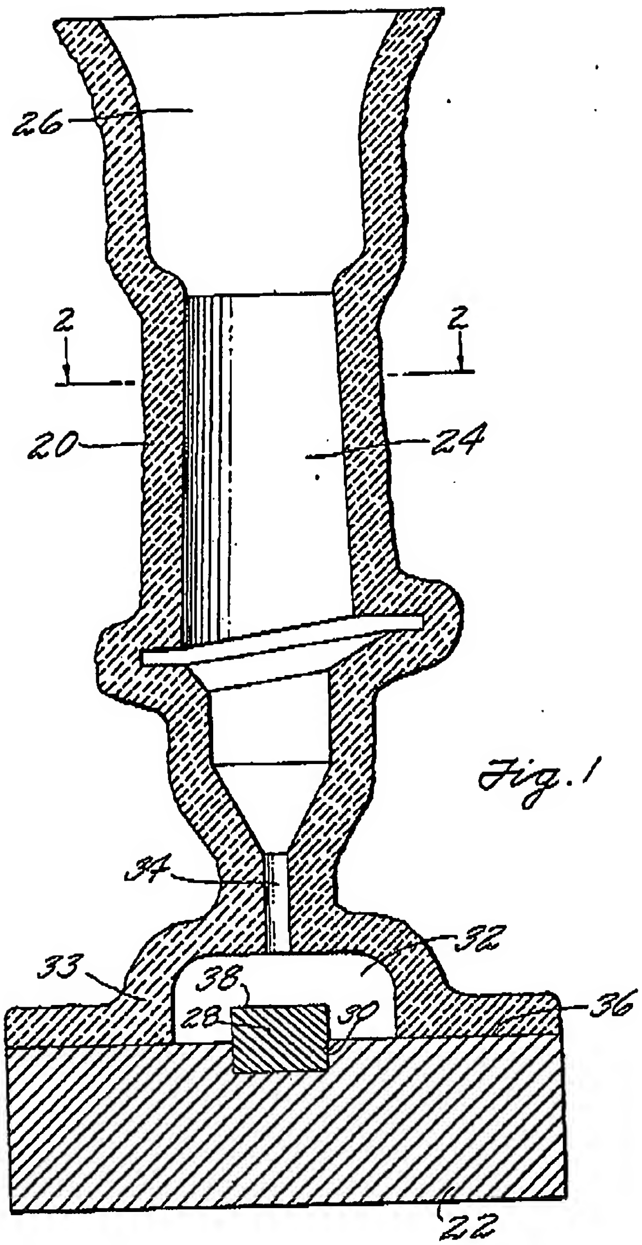

Fig. 1

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Fig. 2

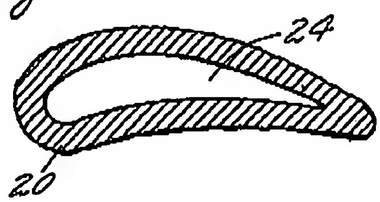


Fig. 3

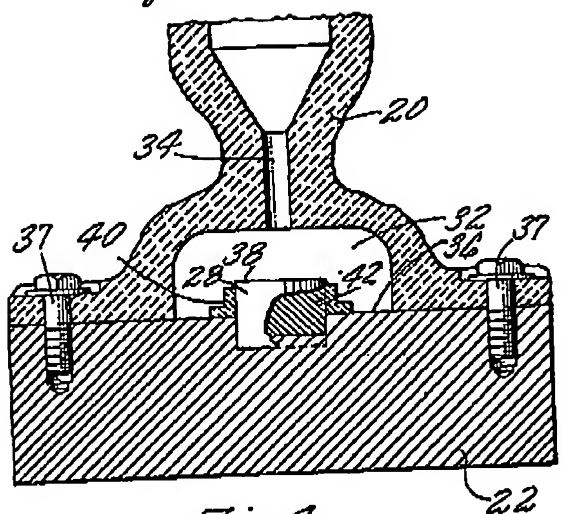


Fig. 4

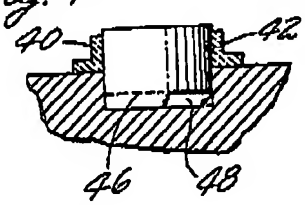


Fig. 5

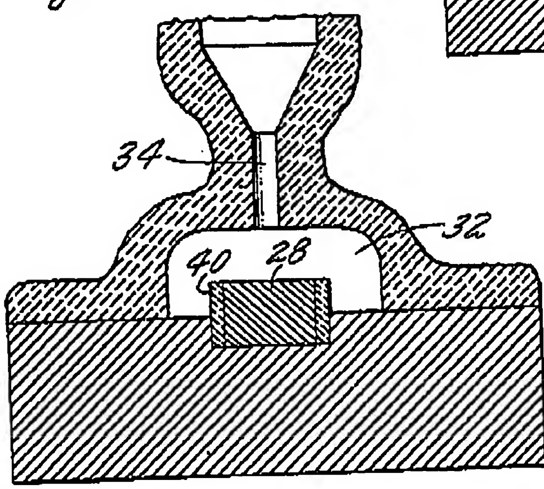
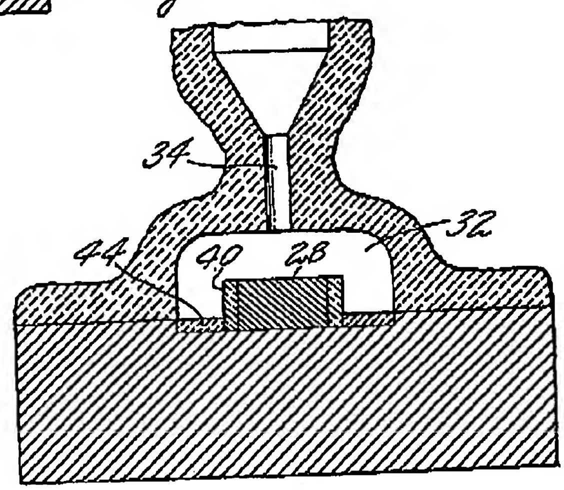


Fig. 6



SPECIFICATION

Method and apparatus for epitaxial solidification

This invention relates to methods and apparatuses for directionally solidifying molten metals, most particularly to the production of single crystals with controlled crystallographic orientation.

It is well known that great improvements in the performance of metal structures can be achieved by unidirectional casting techniques which produce articles with columnar grain or single crystals. See, for example, the teachings of Ver Snyder, U.S. Patent 3 260 505, and Piearcey, U.S. Patent 3 494 709. The principal objective of the prior art apparatus, methods, and articles has been to provide structures which have enhanced properties along the principal axis of the article, that is, the principal axis of the article is typically the solidification growth axis or the axis along which the solidification front is caused to move.

When metals are directionally solidified, they often by nature solidify or grow faster in one crystallographic orientation than others. For example, in nickel base super-alloys the [001] orientation is found to predominate. As a result, single crystal castings made by means disclosed in U.S. Patent 3 494 709, mentioned above, will have the [001] orientation lying along the growth axis. Therefore, to produce another crystallographic orientation along the principal axis of solidification specialized techniques must be used. Also, the orientation of crystals with respect to the plane perpendicular to the axis of solidification is randon in most directionally solidified articles unless steps are taken to achieve control. The crystallographic orientation measured along the principal axis of a casting is called the primary orientation, while the polar orientation in the plane perpendicualr to the principal axis is called the secondary orientation.

The properties of a material such as a columnar grain or single crystal material are influenced by its crystallographic orientation. For example, the elastic moduli will be importantly varied in many alloys and the performance of parts under stress and strain thereby affected. Consequently in more sophisticated applications of advanced materials, it is of increasing importance to control both the primary and secondary orientations. The crystallographic orientations of materials are determinable by conventional nondestructive laboratory techniques. Radiographic diffraction, e.g. by the Laue method, is most useful. Furthermore, changes in crystallographic structure can be readily ascertained by conventional grain etching. If the orientation at a location in a part is determined, the orientation will be the same in another region in the absence of an intervening grain boundary, and absent subtle crystal variations beyond the scope of this discussion.

A useful technique for controlling crystallographic structure in cast articles is the use of a previously made metal seed which has the desired structure. If the article casting can be made to grow epitaxially from the seed, the seed structure will be reproduced.

Of course, growing objects from seeds is well known. For instance the Bridgman method for epitaxial single crystal formation disclosed in U.S. Patent 1 793 672 and other publications dates from the 1920's. Delano in U.S. Patent 2 791 813 describes structures with controlled crystallographic orientations in which seed crystals are used to attain the desired result. Barrow et al in U.S. Patent 3 759 310 describes an apparatus and electric arc method for making single crystal articles with a consumable electrode in which a seed crystal at the bottom of the mold is used. More recently, Petrov et al in U.S. Patent 3 857 436 describes an improved method and apparatus for manufacturing single crystal articles. Disclosed therein are means and methods for initiating crystallization at a conical-shaped bottom chamber where abrupt super-cooling conditions are created. Petrov U.S. Patent describes further refinements. Also, Copley U.S. Patent 3 598 169, discloses the casting of relatively flat objects using seed wedges and accomplishing radially outward solidification.

With the exception of Barrow, all the aforementioned techniques anticipate heating the mold prior to the introduction of the molten metal. The practice in the prior art is that the seed is in the mold during the heating. Therefore, it is also heated with the mold to a relatively high temperature although in some situations its location would indicate lesser heating. As the superheated molten metal is introduced into the mold and allowed to stabilize, it contacts the heated seed and causes it to partially melt. Of course it is necessary to melt at least part, but only a part, of the seed, and this necessitates a control over the initial and transient conditions of the seed, mold, molten metal, and other influential factors.

Much of the prior art reflects laboratory technique and is not oriented toward mass production. Now, there is a trend towards greater commercial utilization of articles having controlled crystallographic structure, such as columnar grain and single crystal gas turbine airfoils. This has impelled the development of automated casting techniques to produce articles in quantity on an economic basis. According to one of these techniques, described in King et al, U.S. Patent 3 895 672, a heated mold is clamped onto a cool chill plate just immediately prior to the introduction of molten metal into the mold. If the seed crystal is used, it is attached to the chill plate and it is, of course, correspondingly cool. The short duration between the mating of the hot mold and the cool chill plate provide little time for the temperature of the seed to increase. The same difficulty can obtain in some of the prior art apparatus and methods. If the seed is too cold, insufficient melting will occur and epitaxy will not result. One method of overcoming this is to increasingly superheat the molten metal but to do so is disadvantageous since superheating often leads to increased time and cost, undesired vaporization of elements, and increased degradation of the mold. To separately heat the seed or to include the seed with the mold when the mold is being heated after the methods of the older art is also disadvantageous, both from the mechanical and manufacturing complications and because the

seed can become unduly oxidized or otherwise contaminated.

Another consideration during the manufacture of articles of controlled primary and secondary crystallographic orientation is that after manufacture, the orientation of the seed must be, first, accurately defined by suitable inspection techniques, and, second, controlled precisely with respect to the axes of the articles being cast. Accordingly, the providing of seeds for casting can represent a significant cost. It is, therefore, desirable that seeds be recovered from the casting process after the article is formed and reused if possible. However, when the seed is severely melted during the casting operation or surrounded by a larger quantity of solidified metal of extraneous orientation, recovery for reuse is difficult.

An object of the invention is to provide an improved method, apparatus, and mold for the production of castings of controlled crystallographic orientation using epitaxial growth from seeds having a known orientation. A further object of the invention is to provide for the preservation, recovery, and reuse of seeds.

According to the invention, molten metal is introduced into a directional solidification mold in a manner which causes a portion to flow over the seed to heat and melt a part of it, and remove any undesirable contamination film which may be present. When used conjunctively with a chill plate, the mold is configured to define a starter cavity of sufficient volume to contain the seed and receive molten metal flowed over the seed. The seed may project into the starter cavity to allow molten metal to flow about and heat it. A barrier layer, such as a ceramic coating, may be provided on selected portions of the seed to facilitate its removal from the solid metal casting for reuse. In one embodiment, thermal insulation is placed on the chill plate in portions adjacent the seed to slow solidification of molten metal of uncontrolled orientation within the starter cavity and ensure that epitaxially solidified metal originating from the seed will be present in the article.

In one embodiment of the invention, a mold has an article section connected to the starter section by a selector section. The starter section is adapted to contain the seed and to provide a volume capable of receiving a portion of the molten metal flowed about the seed to heat and melt it. The selector section is located in close proximity to the region in the starter section where the seed is receivable and functions to only allow metal epitaxially solidified from the seed to pass into the article section. In a preferred embodiment, the mold is adapted to receive molten metal through the article section and its discharge from selector section in which it passes is controlled to impinge on the surface of the seed and thereby effectively heat and melt the seed.

The invention is suitable for the production of cast articles of any alloy, in any desired controlled structure producible from a seed. Of particular useful application is the production of columnar grain or single crystal components of nickel superalloys.

The invention achieves the appropriate melting of the seed to ensure the desired epitaxial growth therefrom, overcoming the defective castings which may be produced when the seed is not adequately melted or the contamination layer not fully removed. Further, the invention allows the use of seed crystals which are not heated substantially prior to the introduction of molten metal into the mold. In a preferred embodiment, it further reduces the cost of seeds by providing for their ready recovery from solidified castings and subsequent reuse. The use of seeds is made more economic and therefore more feasible compared to growth without seeding, allowing the realization of benefits from primary and secondary orientation control. Single crystal mold design can be simplified and initial solidification rates increased, thereby increasing production yield.

The foregoing and other objects, features, and advantages of the present invention will become more apparent from the detailed description of the preferred embodiment and the accompanying drawings which follow.

Figure 1 shows a mold containing a seed mounted on a chill plate in cross section.

Figure 2 shows a transverse cross section of the article cavity of the mold in Figure 1.

Figure 3 is a partial sectional view of a seed cavity of a mold on a chill plate.

Figure 4 is a detail of the seed seating.

Figure 5 is a partial sectional view of a seed with a barrier layer around its periphery.

Figure 6 is a partial sectional view of an alternate embodiment of seed and chill plate barrier layers.

The preferred embodiment is described in terms of a mold particularly adapted to be utilized generally within the teachings of the aforementioned King et al U.S. Patent 3 895 672, for the production of one piece single crystal nickel alloy castings, although its use is not limited to such.

A mold 20 made of a ceramic material suitable for foming a single crystal article is mounted on a copper chill plate 22 as shown in Figure 1. The mold is comprised of a section which defines an article cavity 24 which, as Figure 2 indicates, is configured to a gas turbine airfoil, to the production of which the present invention especially contributes. The mold further has a first end 26 for receiving molten metal and passing it into the article cavity and a second end 33 adapted to contact a chill plate.

A seed 28, having a predetermined crystallographic oreintation, is mounted in a recess 30 in the chill plate 22. The seed is therefore in intimate contact with, and will be cooled by, the chill plte. Surrounding the seed is a starter cavity 32 defined by the second end 33, the starter section, of the mold and the chill plate 22. A selector section 34 connects the starter cavity 32 and the article cavity 24. The selector section 34 has a substantially smaller cross sectional area than either the seed crystal cavity or article cavity.

In the preferred embodiment shown, the seed, starter cavity, and selector section are circular in cross section although other cross section shapes are equally functional.

The relative sizes of the respective elements is not fixed but may be put in general perspective by way

of an example: When fabricating nickel superalloy articles, such a gas turbine airfoils 10 to 25 centimeters high, a seed of the superalloy with a diameter of 2-2.5 cm and a similar height would be preferable. The starter cavity would have a diameter of about 5 cm and the entrance to the selector section would be about 0.5 - 1.0 cm above the surface of the seed. Thus the starter cavity would have a volume of more than five times that of the seed contained therein. As is pointed out below, this volume is available for receiving molten metal for heating the seed and initiating epitaxial solidification therefrom.

The starter section end 33 is placed tightly against the chill plate at its surface 36 to prevent the escape of molten metal. Means for clamping, shown as bolts in Figure 3, are utilized to maintain good contact between the mold and chill plate. Other mechanical fasteners and fixtures are equally suitable so long as they are located out of the molten metal path and are adapted to holding a mold which is at a high temperature. Of course, in mass production, a criterion in the selection of clamping means is the ease and speed of engagement and release.

When the mold and chill plate are firmly clamped together, the assembly is adapted to be placed within various apparatuses described in the prior art for directional solidification. Molten metal can be introduced and the requisite thermal gradient applied to the mold to cause directional solidification of the casting. The use of the apparatus is as follows. Molten metal is introduced into the mold 20 through the receiving end 26, passing thereupon through the article section 24 and selector section 34 and impinging on and flowing across the surface 38 of the seed 28. The action of the molten metal on the seed surface 38 thereby heats it and causes it to melt and through turbulence enhances the removal of any deposits or films. The molten metal having passed across the surface of the seed is deposited in the starter cavity 32 adjacent the seed. Thus the starter cavity functions as a receiving reservoir for the molten metal used to heat the metal. The receiving reservoir could be located apart from the cavity containing the seed, if desired. Metal introduction by a separate gate, as shown in U.S. Patent 3 915 761 is another option. In such cases the starter cavity still must be configured to allow through-flow of molten metal. As the elements are configured in the preferred embodiment of Figure 1, after passage over the seed surface, the molten metal surrounds the seed laterally and thereby further imparts heat to it.

When the mold has been filled with metal, by withdrawal of heat through the chill and mold walls according to known practice, molten metal is caused to solidify progressively along the principal axis of the mold, that is, vertically. Metal in the starter section will solidify first, and of course a major portion of the seed is present as a solid throughout. Inasmuch as the selector section 34 is centered above the seed 28, metal which solidifies epitaxially on the surface of the seed will desirably first reach the selector section and pass therethrough. Since the solidifying metal passing through the selector section solidified epitaxially from the seed crystal, it will have the same orientation as the seed crystal. In like fashion, the article formed in cavity 24 will have a similar orientation, as it takes its structure from the earlier-formed material of the selector section.

Figure 3 illustrates in more detail the arrangement of the important elements of the invention in the starter section. To obtain a desired secondary orientation, it is necessary that the seed crystal be oriented in a predetermined manner with respect to the article cavity 24. This is achievable by orienting both the seed and mold in fixed relationship to the chill plate 22. As shown n Figure 3, the mold is oriented to the chill plate by means of bolts 37 which also have the function of clamping the mold to the chill plate to prevent leakage. Of course, other orienting means can be utilized, particularly in mass production, such as polarizing of the chill plate and mold by shape at their contact points or using electro-optical sensors with suitable indices. Shown in the detail of Figure 4 are means for orienting the seed with respect to the chill plate. Vertical or primary axis orientation is carried out by the obvious means of resting the seed on the surface of the chill plate. The secondary orientation, or the polar orientation about the primary axis, is controlled by means of a mating slot and key. As shown, the seed crystal has a simple slot 48 across its diameter while the chill plate is provided with an integral key way 48. Other mechanical detents and locators and other polarizing methods will also be suitable.

Further shown in Figures 3 and 4 is a ceramic shield 40 surrounding the circumference of the seed 28. This is a barrier layer to prevent molten metal which has passed over the surface 38 of the seed and come to rest in the starter cavity 32 from adhering to the circumference 42 of the seed. The shield 40 will tend to inhibit melting at the seed circumference 42 and will prevent adherence of the molten metal in the cavity to the seed circumference. Accordingly, after the metal in the cavity 32 has solidified and the entire casting is removed from the mold, the casting can be cut across the plane of surface 38 and the seed will thereby be readily detachable from the starter section casting, and with minor preparation can be reused.

Figure 5 shows an alternate embodiment of the ceramic shield 40 wherein the shield is recessed into the chill plate with the seed. The shield can be constructed from a ceramic material or any other substance which is resistant to the action of the molten metal during the short time it is exposed to it prior to solidification. It is only required that the shield be formed of a material which has the requisite thermal and corrosion resistance and is in addition of sufficient mechanical strength to not become loose under the action of the molten metal. Of course to achieve the object of the invention, the barrier layer around the seed circumference need not be a separate physical element but can be a coating on the seed as well. Figure 6 shows a still further embodiment of the invention in which the seed is mounted flush with a depressed region of the surface of the chill plate together with shield 40. Shown in addition is a ceramic annular disc 44 which is resting on the chill plate surface 36 adjacent the seed. The disc 44 has the function of reducing the

cooling through the chill plate, and therefore the rate of solidification of the molten metal adjacent the seed, compared to what it would be if the disc were not present. Naturally, the metal solidifying from the chill plate surface 38 will not have the desired crystallographic orientation of the seed. In particular starter cavity configurations, the presence of the disc 44 gives more assurance that metal having an undesired crystallographic orientation will not reach the selector section 34 before that metal epitaxially solidifying from the seed surface 38. In Figure 6, the disc 44 is shown as a separate element covering the entire exposed chill plate in the cavity 32. However, the diametrical extent of coverage can be varied, for example, by decreasing the diameter of the disc 44 so that some of the chill plate surface at the periphery of cavity is exposed. Variation of the coverage of the chill plate would controllably vary heat extraction from the metal in cavity 32 to effect the desired solidification of the article. In addition, the disc 44 may be made integral with the shield 40 as is shown in Figure 3. As another alternate, the disc 44 can be made integral with the mold 20, in which case the inner diameter of the disc portion would be varied to control heat extraction. The disc 44 can also be configured as a coating on the chill plate, and the functioning of the disc can be varied by the thickness and thermal characteristics of the material of construction.

The use of the apparatus and method described herein can be adapted to the production of single parts or multiple parts. Of course, multiple pieces can be made by arranging a multiplicity of molds of the type shown in Figure 1 as an assembly, as is the common practice in the mass production of directionally solidified investment castings. Alternately, more than one part may be made from a single seed crystal by spreading the mold immediately above the selector section, somewhat in the manner of Petrov, U.S. Patent 3 857 436.

While the foregoing invention has been described in the preferred embodiment in terms of a single crystal casting, it is within the contemplation of the invention that columnar grain castings and other epitaxially derived casting structures will be produced. The invention is usable with any castable alloy for which a suitable mold can be fabricated. It will further be understood by those skilled in the art that various changes and omissions in the form and detail thereof may be made therein without departing from the scope of the invention defined in the appended claims.

CLAIMS

1. An apparatus for solidifying molten metal into an article having controlled crystallographic orientation, characterized by:

a chill plate for cooling molten metal during directional solidification;

a seed of a known crystallographic orientation for initiating epitaxial solidification which is desired in the article;

means for holding the seed in predetermined orientation with respect to the mold in proximity to the chill plate so that heat transfer from the seed to the chill plate can be achieved;

a mold having an article section and a starter section shaped to cause flow of molten metal about the seed, the starter section being in contact with the chill plate, and conjunctively with the chill plate defining a volume capable of containing the seed; and

means for holding the mold fixedly to the chill plate, to control the orientation of the mold with respect to the seed.

2. The apparatus according to claim 1 characterized in that the mold has a selector section connecting the article section and the starter section to ensure that only epitaxially solidified material is formed in the article section.

3. The apparatus according to claim 1 characterized in that means is provided for impinging molten metal on the surface of the seed so that heating is increased and surface contamination films which interfere with epitaxial solidification are removed.

4. The apparatus according to claim 1 characterized in that it further includes a seed which projects into the starter cavity so that molten metal flowing into the starter cavity surrounds the seed at a surface other than that from which epitaxial solidification into the article takes place, to provide additional heating to the seed.

5. The apparatus according to claim 2 characterized in that means is provided for preventing molten metal from adhering to the seed at predetermined location which facilitate the removal of the seed from the solidified casting.

6. The apparatus according to claims 1 or 2 characterized in that means is provided for thermally insulating a portion of the chill plate adjacent the seed to lessen heat loss from surplus metal placed in the starter cavity.

7. The apparatus according to claim 1 characterized in that the seed is a single crystal.

8. A method of casting metal into an article having controlled crystallographic orientation with the apparatus according to any of claims 1-7 using epitaxial directional solidification from a seed which is initially substantially cooler than the melting point of the metal being cast, characterized by:

impinging and flowing molten metal over the surface of the seed in sufficient quantity to heat and melt a portion of the seed and remove surface contamination which interferes with epitaxial solidification from the seed.

9. The method of casting metals into an article having controlled crystallographic orientation, according to claim 8 characterized by:

placing a seed on a chill plate in a controlled orientation thereto;

providing a heated mold on the chill plate in a fixed orientation thereto to contain the seed therein and receive molten metal:

filling the mold with molten metal using a means which ensures that a portion of the molten metal flows across the surface of the seed to a reservoir in sufficient quantity to heat and partially melt a portion of the seed and remove any contamination films thereon; and

epitaxially solidifying the molten metal to form an article from the seed having a crystallographic orientation determined by the seed.

10. The method according to claim 9, characterized by:

placing a barrier layer resistant to the molten metal around a portion of the seed for preventing adherence of molten metal to the seed, without eliminating the envelopment of the seed by molten metal which would occur in the absence of the barrier layer, to facilitate removal of the seed from the casting after solidification.

11. The method according to claim 8 of casting metals into an article having controlled crystallographic orientation, characterized by:

forming a mold which has a first end adapted to receive molten metal, a second end adapted to define a starter cavity when in contact with a chill plate, an article cavity adapted to contain and form molten metal and a selector section located between the second end and the article cavity;

heating the mold;

providing on a cool chill plate a seed substantially smaller than the starter cavity;

contacting the second end of the mold with the chill plate in a manner which both contains the seed in the starter cavity and provides a space within the cavity adapted to receive and contain molten metal;

filling the mold by pouring molten metal into the first end of the mold, so that the molten metal passes to the second end and flows to a reservoir by passing across the surface of the seed, to heat and melt a portion of the seed:

cooling the mold so that the molten metal solidifies epitaxially from the second end into the article cavity to form and article having a crystallographic orientation determined by the seed.

12. A mold for epitaxially casting metal from a seed into an article of cotrolled crystallographic orientation wherein the mold is fixed to a chill plate and molten metal is directionally solidified, characterized by:

an article cavity for forming molten metal into an article;

a selector cavity connected to the article cavity for controlling the crystallographic orientation of metal which grows by solidification into the article cavity from a starter cavity;

a starter cavity connected to the selector cavity, having a cross sectional area substantially greater than the selector cavity and a volume sufficient to contain a seed and molten metal introduced into the mold for heating a seed, and;

means for introduction of molten metal into the mold so that it flows across the seed to the starter cavity where there is a volume to contain the molten metal.

13. The mold according to claim 11 characterized in that the selector cavity is a substantially straight channel aligned between the starter cavity and the article cavity, the channel axis being substantially parallel to the principal growth axis of the article formable in the mold.

14. The mold according to claim 12 characterized in that the mold starter cavity is shaped to minimize heat loss to a chill plate in regions adjacent the seed.

15. An apparatus for solidifying molten metal into an article having controlled crystallographic orientation as hereinbefore described with reference to the accompanying drawings.

16. A method of casting metal into an article having controlled crystallographic orientation as hereinbefore described with reference to the accompanying drawings.

Printed for Her Majesty's Stationery Office by Croydon Printing Company Limited, Croydon Surrey, 1980.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.